
Separation of Food Colorings via Liquid–Liquid Extraction: An At-Home Organic Chemistry Lab

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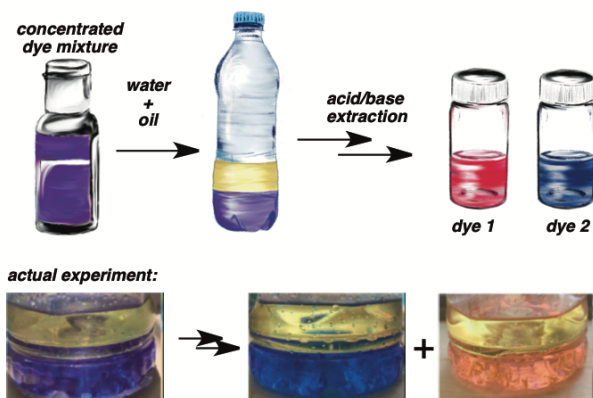
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ABSTRACT

Herein, we describe an accessible and safe organic chemistry lab experiment that can be completed at home. Liquid–liquid extraction is a fundamental organic chemistry lab experiment that touches on topics such as chemical structure, density, solubility, and acid–base chemistry. Given the increased demand for safe organic chemistry experiments that can be performed in the at-home environment, we have adapted the conventional wet lab liquid-liquid extraction experience by using food colorants. Students are first guided through sample questions to establish a basic understanding of solubility, acid–base chemistry, and separation via extraction techniques. Next, they are given unknown dye mixtures which they subject to liquid–liquid extraction using a plastic soda bottle with vegetable oil, water, white vinegar, and sodium bicarbonate. All materials are readily available and food safe, making the experiment amenable to the at-home environment while still allowing students to physically engage in a foundational lab experiment.

GRAPHICAL ABSTRACT



KEYWORDS

Second-Year Undergraduate, Organic Chemistry, Laboratory Instruction, Hands-On Learning/t, Acids/Bases, Extraction, Dyes/Pigments, Physical Properties, Separation Science

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INTRODUCTION

Liquid-liquid extraction is a conceptually challenging lab technique often introduced in the first semester of the organic chemistry lab sequence. While the procedure itself is of moderate complexity to the novice organic chemistry student, the underlying concepts and their interconnectedness provide a more nuanced opportunity to probe student understanding of solubility/miscibility, density, and acid-base chemistry. In the classic liquid-liquid extraction separation procedure, two components (or more) are separated by using base to selectively deprotonate one component rendering it soluble in water while the other component remains soluble in the organic phase.¹⁻⁷ After separation of the phases, the aqueous phase is then acidified, reprotonating the water soluble conjugate base to yield an insoluble material that can be removed by filtration or by extraction with an organic phase.

Alternately, a component that readily protonates may be extracted into an aqueous phase using acid, and then base to return the component to the organic phase. Many adaptations have been made to the standard liquid-liquid extraction procedure, including the use of vibrant dyes⁸⁻¹² to aid in visualization of the acid-base chemistry taking place as well as approaches that do not rely on wet lab experiments¹³ to help students understand specific concept areas (acid-base chemistry, density, etc.).

Herein, an at-home experiment to separate food colorings via liquid-liquid extraction is described. The experiment corresponds to one 2-4 hour laboratory period, depending on how many samples are examined and whether any questions or analyses are incorporated. Typically, this experiment would be one out of 10-12 experiments conducted in the first semester organic chemistry laboratory. Students commence by completing a prelaboratory assignment that includes videos or readings on standard extraction techniques using separatory funnels and how acidity/basicity drive solubility. After reading the laboratory directions, researching the relevant dye structures, and writing an experimental outline in their laboratory notebook, students then undertake the experiment at home using readily available materials. After the experience with the practical portion, they then complete a

theoretical exercise to separate a set of simple organic molecules by means of acid-base chemistry in combination with extraction.

DESIGN OF EXPERIMENT

At-Home Considerations

The primary drivers in design of this at-home experiment were 1) safety, 2) availability of materials, and 3) providing an equivalent experience (in terms of both the physical experience and in probing the underlying theoretical concepts).

The use of safe, easily available feedstocks is especially important for an at-home chemistry lab as access to safety resources found in the undergraduate organic chemistry lab are absent. Students performing at-home lab experiments should be provided with the safest possible materials that deliver the same pedagogical outcomes as a conventional lab setting. Thus, we elected to use all food-safe materials in this experiment. Despite the use of chemically benign materials, there is still room for discussion surrounding safety and chemical hazards relevant to in-person instruction. In this instance, students were required to complete safety training including a quiz in an earlier portion of the course. PPE in the form of gloves and eyewear along with a safety assessment were provided.

Instructors wishing to include this experiment in an at-home lab curriculum should consult with the governing body on environmental, health, and safety at their institution to ensure appropriate compliance. In our case, the department at the University of Pennsylvania that monitors lab safety reviewed the protocol and materials. Students needed to sign a safety agreement (see Supporting Information). Moreover, students, if conducting experimentation in a place in which they are not the homeowner/primary lessee, were asked to fill out a space agreement to ensure proper safety in the at-home lab (see Supporting Information).

Due to use of only food-safe materials, supplies can 1) be sent to students in a pre-made kit assembled at the home institution, 2) be sent to students through a chemical supplier that makes kits, or 3) be easily purchased by the student at a local grocery store. A hybrid approach can also be utilized in which students are sent unknown food coloring mixtures and are responsible for providing cooking oil, white vinegar, and baking soda. In the case where option three or a hybrid approach is utilized, instructors should ensure that students enrolled in the course have access to the monetary

resources required to purchase materials. In the case that a student demonstrates financial need, a plan should be in place at the onset of the course to assist any students needing financial assistance to obtain materials required for the successful completion of the practical portion of this experiment.

In lieu of a separatory funnel, this experiment uses a 4-8 oz clear plastic bottle to accomplish the liquid-liquid extraction. To ensure that students retain exposure to the relevant laboratory glassware and techniques, videos of extractions with separatory funnels and standard laboratory glassware were incorporated in the prelaboratory segment. A wide variety of virtual lab platforms allow students to engage with specialized glassware and apparatuses used in the university lab. Moreover, the motor actions required to complete this experiment closely emulate those used in a standard liquid-liquid extraction protocol.

Properties of Dyes, Selection of Dyes

The specific dyes used in this experiment were selected based on availability and the ability to partition into different layers based on the functional groups present. Most food coloring dyes contain arylsulfonic acid salts that will not protonate in the presence of a mild acid such as acetic acid. On the other hand, the fluorescein dyes contain phenolate and carboxylic acid salts that will protonate in the presence of excess acetic acid and undergo a change in solubility. Notably, protonation of fluorescein also results in quenching of the color due to cyclization of the dye to a colorless form (Figure 1). As such, fluorescein dyes are not “color-fast”, meaning they change color in response to changes in pH, whereas most other food dyes are color-fast.

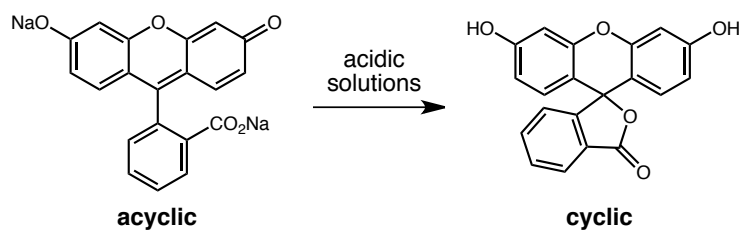


Figure 1. Structure of fluorescein under neutral/basic vs acidic conditions.

In this example, red dye #3, a spirocyclic carboxylic acid which is only chromophoric under neutral or basic conditions, is utilized as the pH-sensitive component (Figure 2). Other fluorescein dye derivatives such as acid yellow 73 could be utilized in this experiment; however, they are not as readily

available or commonly used in food preparations. The use of fluorescein dye also provides an option to discuss higher level topics for the advanced organic chemistry student such as conjugate addition reactions and pH-triggered reactions. Both blue dye #1 and red dye #40 are colorfast and do not change color with changes in pH.

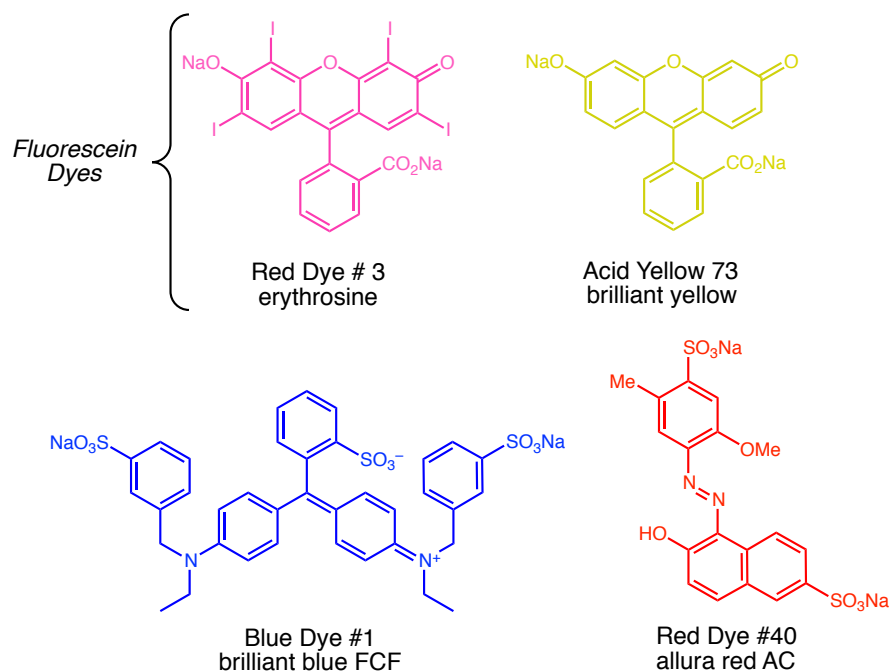


Figure 2. Structures of different food coloring dyes.

Despite the focus on a few dyes here, many other food coloring dyes including complex mixtures exist and could be substituted into this experimental protocol. However, careful attention to their safety, availability, aqueous/organic solubility, and pH sensitivity should be made before use by students in an at-home lab. Bulk food colorings are typically supplied as aqueous solutions, but some food colorings are rated as either water or oil soluble dependent upon their intended application in food preparation. Most often, this solubility is controlled by additives rather than by protonation state, but care must be taken in selection of both the specific dyes used and their formulation. Here, liquid food colorings (super red, rose pink, royal blue, and violet) from U.S. Cake Supply (Food Grade, 0.75 fl oz = 20 mL) were employed. Less than one-half of the bottle was sufficient to supply material for 40 experiment kits.

Selection of solvents

As previously mentioned, safety was the top priority in designing this experiment. There were no issues with the use of water for the aqueous layer, but safety considerations excluded many solvents that would typically be used in extraction chemistry (e.g., ethyl acetate, dichloromethane, etc.). While it is possible to ship small quantities of such solvents, there are significant regulatory hurdles and the use of such solvents in the less well-controlled at-home setting was undesirable due to their toxicity and flammability. In addition, many kitchen fixtures are not resistant to such solvents (countertops, tables, etc.). Thus, we limited our pool of potential extraction solvents to materials available at retail suppliers accessible to the public. The obvious choices for versatile organic solvents readily available to the everyday consumer would be acetone, isopropanol, or ethanol. The first is undesirable due to its miscibility in aqueous media. Although isopropanol can be partitioned with water by salting-out, this added complexity was undesirable in this particular experimental design. Finally, ethanol presents all of the above pitfalls and is also regulated at higher concentrations. Thus, our experimental design used an unconventional organic phase, namely cooking oil.

Classic elementary chemistry demonstrations rely on the immiscibility of water and oil to demonstrate density and solubility in an easy-to-digest visual. Moreover, some of these demonstrations incorporate the use of colorful dyes to help onlookers better visualize the biphasic system created upon mixture of water with oil. In addition to being safe (nonvolatile, low flammability relative to organic solvents typically used) cooking oil is ubiquitous. It is important to use a lightly colored oil (i.e. pale yellow) such as vegetable, canola, or safflower oil, to ensure that students can easily visualize the partitioning of the food colorings. Darkly colored oils such as olive, sesame, and avocado oil should be avoided as should oils with added flavorings or colorants. Typically, oil partitions well from water even after vigorous shaking, but instructors may prefer to use “gentle shaking” or “gentle mixing” to limit emulsions.

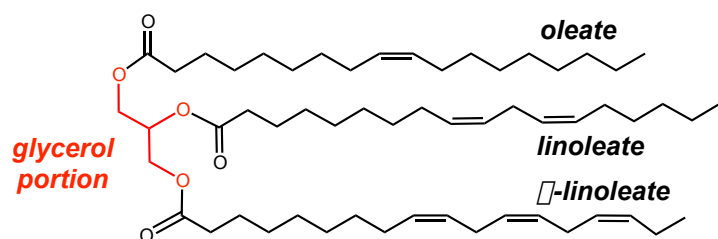


Figure 3. Structures of oleic and linoleic acid.

Cooking oils are mainly comprised of triglycerides which are esters of glycerol and oleic acid/linoleic acids (Figure 3).¹⁴ Students can become confused about the properties of triglycerides and additional background can be included to mitigate these concerns since online searches focus on the acid portions of the structures. Vegetable oils are largely neutral, but can contain small percentages of the free fatty acids. On their own fatty acids are not acidic, since this acidity requires the presence of water. Cooking oils have low solubility in water and water has low solubility in cooking oils. When exposed to neutral or acidic aqueous layers, cooking oils do form a separate organic layer. If free fatty acids are present in the cooking oil, they can deprotonate under basic conditions. For example, oleic acid has a pK_a of 5.02.¹⁵ When deprotonated, the resultant compounds are soaps exhibiting mixed solubility properties and may give rise to emulsions. Extended treatment with strong bases (NaOH, Na_2CO_3) can even cause hydrolysis of the triglycerides.

Another option is to use mineral oil,¹⁵ a mixture of higher alkanes, which is also broadly available in the form of pure mineral oil or baby oil. Care should be taken in checking labels, especially of the latter to determine content. Representative trials have shown that both provide largely the same results as with cooking oil. Laboratory grade mineral oil give more rapid separations than cooking oil while baby oil performs about the same as cooking oil.

Acid-Base

The last consideration in experimental design was the selection of the acid and base for the extraction procedure. In accordance with our safety goals, we chose white vinegar as the acid component and baking soda (sodium bicarbonate) as the base component. With the dyes selected, excess acetic acid is a sufficiently strong acid to protonate the fluorescein-based dye changing its solubility. Similarly, use of excess baking soda is sufficiently strong to deprotonate the -OH functionalities present in the chosen dyes.

OVERVIEW OF ACTIVITY

Prelab Lecture Materials

Before the experiment is given, students received a combination of synchronous instruction and asynchronous engagement. Students had readings,¹⁶ lecture videos,¹⁷ and guided online problems to build an understanding of the fundamentals: organic vs aqueous phase, density, miscibility. A review

of acid-base chemistry was also provided. Students further needed to understand the solubility of
175 different protonation states of compounds with different pH-sensitive groups, which is more
conceptually advanced than introductory acid-base chemistry. Finally, students were guided through
a case study: the separation of benzoic acid, naphthol, and naphthalene by means of successive
extractions from a diethyl ether solution with aqueous sodium bicarbonate and aqueous sodium
hydroxide.¹⁷ Subsequent acidification of the individual aqueous portions allowed isolation of each
180 neutral compound via precipitation or extraction into a further organic phase. Depending on
placement in the sequence, the separated compounds could be further isolated by drying over a
chemical desiccant, filtration, rotary evaporation, and recrystallization.

By the end of the prelab session, students had been exposed to the following topics:

- Extraction technique
 - 185 • Organic/aqueous phase
 - Density
 - Like dissolves like
- Acid/base chemistry
- Solubility of different protonation states
- 190 • Separation

Moreover, students were adequately primed to complete the prelab write-up which required them
to create a flowchart of the various potential outcomes when mixing each dye with acid or basic
solutions.

Prelab Write-Up

195 Before the experiment was conducted, students were provided the names of the possible dyes in
their food coloring samples. Students then researched the dye compounds under consideration with
the goals of identifying their chemical structures and the pK_a of the pH-sensitive groups present in
each molecule. Some online resources provide the pK_a of the “overall” chemical structure, which led
students to erroneous conclusions. Thus, it was important to direct students to identify specific pK_a
200 values of each acidic functional group identified. These values could be found by more detailed
internet searches or by directing students to generic tables and asking them to find the closest
matching acidic functional group. Using this information, students were directed to predict which

compounds would be in which layer under neutral, acidic, or basic conditions (see below). The overarching principle is that neutral organic compounds should be soluble in organic phases and charged organic compounds should be soluble in aqueous phases. The limitations of these concepts need to be addressed in the prelab lectures (e.g. very hydrophobic charged species may be soluble in an organic solvent). The students provided a write-up in their lab notebooks describing the purpose of the experiment, the above structures and pK_a values, a proposed flow chart for their separation, and a procedure (set of steps summarizing, but not duplicating the directions) to follow as they conducted the experiment. These materials were collected and assessed before the experiment was begun to ensure safe procedures and to provide feedback.

An example of experimental objectives and a flow chart are shown in Figure 4. Specifically, they needed to indicate what would happen to a neutral aqueous mixture of red dye #40/blue dye #1 or red dye #3/blue dye #1 when subjected to 1) extraction with oil, 2) treatment of both layers with white vinegar (a source of acetic acid), and 3) separation of the resultant layers followed by treatment of each with sodium bicarbonate solution (baking soda).

The players: Red Dye #3 Red Dye # 40 Blue Dye #1

- 1) Can you figure out which red dye is in "rose pink" food coloring?
- 2) Can you tell which of two food colorings (B vs V) has more red dye vs blue dye?

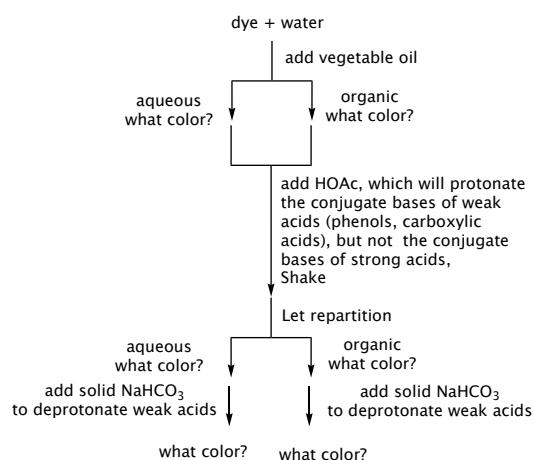


Figure 4. Flow chart of the experiment.

It was also helpful to have a synchronous session to discuss the initial plans outlined in their lab notebooks and resolve questions. After receiving feedback and revising their lab notebook entries as

needed, students then undertook the experiment recording observations in their lab notebooks and taking pictures of different endpoints.

225 Lab

In our exercise, students were given sets of unknown dyes to subject to extraction. Depending on the educational objectives, pure samples could be provided to establish the behavior of the individual dye components which allows students to generate hypotheses for specific mixtures that can be tested or to identify dye components by experimentation. Alternately, the same exercises could be
230 undertaken with only dye mixtures as either unknown (goal = identify dye components) or knowns (goal = predict behavior). In the experiment outlined here, students were presented with dye mixtures that could possibly contain the following food colorings: red dye #40, red dye #3, and blue dye #1. The goals were to 1) identify whether the red colorant was red dye #40 or red dye #3, and 2) to qualitatively determine which of the two dye mixtures had more of the red dye component in it. A range of other
235 food colorings can also be employed.

For this experimental portion, students utilized mixtures of food grade dyes, vegetable oil (organic layer), water, white vinegar, and sodium bicarbonate to perform a safe extraction procedure. The use of food coloring dyes allowed easy assessment of which layer contained the compounds under different conditions.

240 Analysis of Lab

After completing the practical portion of the experiment, students were asked to engage with the material through a series of guided questions that assess their successful integration of the prelab material with the learning outcomes of the experiment. Students were asked to provide their observations of outcomes when their dye mixtures were subjected to the extraction protocol described
245 below. As an added measure to ensure participation and to allow asynchronous assessment, students were asked to supply photographs of the separation apparatus upon:

- Initial mixture of the oil, water, and dye(s) *before* shaking
- Initial mixture of oil, water, and dye(s) *after* shaking
- Mixture of oil, water, and dye(s) after shaking with white vinegar (acetic acid)
- 250 • Aliquot of aqueous phase after mixing with baking soda (sodium bicarbonate) to form a paste

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- Aliquot of organic phase after mixing with baking soda to form a paste

Students were asked to draw the structures of each dye used, indicating their protonation state when treated with white vinegar. They were then asked to then draw the protonation state after the white vinegar solution is treated by sodium bicarbonate. For the dye mixtures (only known to the instructor), students were asked to identify the components and provide an explanation as to why they thought the mixture contains the components proposed. In our version of the experiment, students were also asked which unknown mixture contained more of the red dye and to provide an explanation as to how they had made this determination.

Theoretical Exercise

In the theoretical exercise, students needed to assess pK_a and solubility under different conditions to generate a separation tree (as performed in the prelab) for mixtures of 2-3 compounds; examples included benzoic acid/naphthol/naphthalene, naphthol/para-methoxyaniline/biphenyl, benzylamine/para-methoxyaniline/anthracene, etc. The mixtures were similar to those that would be presented in the lab setting and required a more nuanced understanding of acid-base chemistry than the practical portion of the experiment provided. Any combination of acid/base/neutral compound can be used with various levels of difficulty (i.e. two bases present but one with functionality that is less basic than the other).

In the separation flow chart, students were asked to show ALL steps including extraction with specific solvents, separation of layers, drying (over an anhydrous salt), filtration, solvent removal, distillation, recrystallization, etc. This exercise encouraged the use of pK_a estimates, reported solubilities, boiling point values, and melting points to inform decisions. If a literature source was used, the work had to be cited. An added benefit to this exercise was the incorporation of instruction on chemical information/searching the chemical literature for relevant data.

MATERIALS

The materials required for this experiment were readily accessible. In our case, we provided the starred items. For use elsewhere, these items can either be provided to the students via shipment or obtained from most commercial retailers. Students will need:

- *Safety glasses (not needed given the hazards involved, but good practice)

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- *Plastic gloves (dishwashing gloves or disposable gloves are both acceptable)
 - Clear plastic bottle (makeshift separatory funnel), used water or soda bottles work well
 - *Eye dropper/plastic pipet/straw (to obtain aliquots of the aqueous/organic phase)
 - Straw-colored oil (organic phase substitute; vegetable, canola, safflower, etc.)
 - White vinegar (acetic acid as the source of acid)
 - Water
 - *Food coloring (individual components and/or mixtures)
 - Baking soda (sodium bicarbonate as the source of base)
 - Two bowls/cups (preferably white for color contrast)
 - *pH strips
 - Measuring cups/spoons

EXPERIMENTAL PROTOCOL

After approval of their prelab, students began the hands-on portion of the lab at their own pace. Students first added water to the portion of food dyes (1 drop) provided to them in 1-dram vials. They then transferred the dye solution to a larger container and added one cup of water (dilution to a total volume of 1 cup also works). Students then added $\frac{1}{4}$ cup (60 mL) of this diluted solution to their extraction bottle. Dilution of the dye stock ensured that if a mistake is made in a later step, students had the remaining dye solution ($\frac{3}{4}$ cup) to retry the experiment. Thus, they were encouraged to retain all materials until successful completion of the experiment and the connection was made as to how this approach is standard practice in research laboratories. Vegetable oil was then added to the extraction bottle upon which students were asked to make an observation and took their first of five photos (see above). The bottle was then shaken vigorously, set down, and allowed to partition into two layers (may take up to 20 minutes). Students then recorded the color of the two layers and took their second of five photos. Then, using an appropriate measuring spoon, 2 tsp white vinegar were added to the extraction bottle which was then shaken *gently*. In an optional step, the aqueous layer could be tested with a pH strip to ensure sufficient acidity. If the pH strip indicated the solution was not sufficiently acidic, students added more white vinegar until it was sufficiently acidic. The final amount of white vinegar added and any color changes were noted. Next, the extraction bottle was shaken vigorously, allowed to separate, and the students were directed to take the third of five photos.

In order to probe the response of each layer to base, students used the provided plastic pipet (in this case a 3 mL pipet) to remove a sample of the aqueous layer (1-2 tsp, 5-10 mL) and transfer it to a small white bowl or mug. The procedure was repeated for the organic layer using a second white bowl or mug. To each of the bowls was added baking soda until a paste is formed; amounts of baking soda employed varied depending on how much liquid was sampled, but typically were on the order of 2-4 tsp. Students were warned that fizzing might occur during this step. The paste was mixed with a spoon and the colors of both aqueous and organic paste were recorded in the final two photos. See Figure 4 for a flow chart corresponding to these directions.

We note that there is an alternative protocol possible that mimics a conventional extraction to a greater degree; however, this alternate is more time consuming and requires more containers. After treatment of the dye/oil/water mixture with acetic acid and separation of the layers, it is possible to pipet the two layers into separate containers. Basification of the separated aqueous layer could then be taken in solution format (vs as a paste as we describe). The separated organic layer could be extracted with another aqueous basic portion (baking soda + water) and any deprotonated dyes would move from the organic layer back into the aqueous layer. This approach would be preferable if the experiment is undertaken in a conventional organic laboratory using a separatory funnel. In this context, the experiment represents a particularly green approach to the classic organic chemistry experiment, generating easily disposable, non-hazardous waste.

In our iteration of this experiment, students were provided with 3 dye samples. The first dye sample contained red dye #3 (they were asked to determine if this dye is red dye #3 or red dye #40). The other two samples contained mixtures of red dye #3 and blue dye #1 in differing amounts. In total, the students were asked to conduct the experimental protocol with a total of three samples and to provide the same five photographs for each dye sample giving a total of 15 photographs that were included the lab report. At the end of the experiment, students were asked to take a picture of their hands (palms up, fingertips visible) next to their lab notebook entry with their experimental data and to provide this photograph in their report. This picture was used to assess proper technique (use of gloves). For our lab course, 2/30 students had visible dye on their hands indicating inadequate safety

335 technique. To complete the lab, students were directed to flush all aqueous waste down the drain while handling the oil/oil paste in the same manner as used for cooking oil/grease.

HAZARDS

There were few hazards associated with this experiment as careful attention was taken in ensuring all materials used were food safe, readily available, and easily disposable. Despite this, we built in
340 protocols to this experiment to ensure students were practicing to safe lab technique, despite being at home.

Each student was encouraged to perform this experiment in a well-ventilated area with plenty of space (e.g. a large room, under the stovetop (often with exhaust), on a porch/outside, etc.). There was no harm in performing this experiment in a bathroom or other smaller space, however, a focus on
345 proper lab safety was important if students are to ever use these skills outside of the at-home lab. Moreover, students were asked to wear goggles/safety glasses and gloves which were provided to them in their lab kits. At the end of lab, students were asked to submit pictures of their hands as a way to show they were wearing gloves—each of the dyes used stain skin.

350 RESULTS AND DISCUSSION

This experiment was carried out in a summer 2020 organic chemistry course that required viewing of asynchronous lectures (background and conceptual material) and attendance of a synchronous lecture that reviewed the concepts before implementation (prelaboratory lecture). While students had access to the lab directions and prelab materials before the lecture, they were encouraged to perform
355 the actual hands on experimental portion after the lecture when teaching assistants were available to field potential questions during their work. Thus, the actual lab experimentation done by students was performed asynchronously which required alignment of office hours of the course instructors.

This at home lab experiment was successfully completed by all 30 students in the lab section who were sent the dye mixtures and pipets. The synchronous prelab lecture was approximately 1 h
360 including questions. The prelab write-up was designed to take ~1.5 h. The experimental portion of the lab requires approximately 3-4 h. A detailed procedure can be found in the Supporting Information.

All students (30/30) identified the dyes used and which of the two unknowns had more red dye. With respect to the photos of the extraction experiments, all (30/30) generally achieved the expected outcomes. After initial mixing of the layers and separation (before acidification), the organic portions
365 should have been colorless in all cases. All students did observe more color in the aqueous layer; however, 13 out of 30 had some degree of color bleed through into the organic layer. It appears that this phenomenon is largely due to incomplete separation of layers as the aqueous layers were also cloudy indicative of partial emulsions. The exact type of oil employed (oils supplied by students) and the degree of mixing will influence these outcomes. The oils showing the most bleed through were
370 soybean (5 cases) and canola (8 cases).

In the subsequent theoretical exercise, 25 students (83%) provided a correct separation tree diagram for the extraction of a mixture of three unknowns. The five remaining students did demonstrate partial mastery of the key concepts (e.g. correct separation of two out of three of the compounds). In a later assessment, students were asked to determine the outcome upon extraction of
375 an ethereal mixture acid of yellow 72 (brilliant yellow) and indigo carmine (brilliant blue) with aqueous aqueous acetic acid (69% correct) or aqueous sodium bicarbonate (83% correct).

Finally, at the end of the semester, students were asked to fill out an optional survey in which they ranked which lab they enjoyed most throughout the course. Students were asked to rank where 1 = most favorite lab and 5 = least favorite lab. Of the 28 students who responded to this survey, 54%
380 (15 students) ranked this lab as their favorite and 25% (7 students) ranked as second most favorite, 4% (1 student) ranked as number 3 and, 18% (5 students) ranked as number 4. No students ranked this lab as their least favorite. Overall, this laboratory was highest ranked by the students compared to four other at-home labs (distillation, recrystallization, TLC, and isolation of caffeine).

Although the experimental design described (see below) probes conceptual understanding of acid-
385 base chemistry as well as the general techniques required for separation science, the at-home lab misses some of the technical skill typically gathered in the university setting. For example, students become technically competent in the use of a separatory funnel, something valuable for second semester organic chemistry lab and potential future lab work. Moreover, students may miss out on the collaborative aspect of the university lab experience as they are not as easily able to converse amongst

each other in scenarios require troubleshooting. While this might take away from portions of the conventional learning objectives, the latter concern can be, at least in part, mitigated by encouraging/requiring students to create small groups in which they use video conferencing software to virtually perform the practical portion of the lab together. Moreover, a bounty of virtual, online lab simulators exist in which students can engage with simple to advanced organic chemistry lab technique without the need to be present in the laboratory.

Although the design is not a perfect replica of the in-lab experiment, students should still be able to obtain hands-on experience that will assist in future lab work and their conceptual understanding of separation science.

CONCLUSION

Given the successful completion of this lab by all 30 students, the retention of conceptual knowledge by 76% of the class in a later assessment, the safety, and the simplicity of this experiment, we consider the outcome generally successful. Students were more engaged because of the visual aspects⁸ with food coloring and use of materials from daily life. Based on qualitative comparison to cohorts from the past 11 years that did in-person experiments, this experiment provided equivalent or superior conceptual development without the need for lab space/glassware/organic solvents. Moreover, this lab maintains the hands-on aspect of teaching labs that may be lacking in other virtual or paper-based extraction activities.

In future iterations of this experiment, a synchronous experimentation portion utilizing a virtual meeting platform that allows for separation of students into small groups may be useful. This would strengthen the design of this experiment by re-introducing the collaborative aspect of in-person experimentation. The TAs encouraged students to form small groups to perform the experiment together and reported that some students did take up this opportunity for a more collaborative working environment.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI:

10.1021/acs.jchemed.XXXXXXX. One document containing 1) experimental protocol, 2) safety
420 considerations, 3) distance learning agreement.

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